

# Stress- and Temperature-Dependent Wideband Fluorescence of a Phosphor Composite for Sensing Applications

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**Abstract** As both stress and temperature can alter the lattice of a solid phosphor in a similar way, the dopant rare-earth ion may be used as a probe for either parameter using the same sensing mechanism. Here, a wideband phosphor, YAG:Ce is used as a micro stress sensor at very low magnitudes of compressive stress. The sensing resolution can be smaller than  $0.06 \text{ N/mm}^2$  in the range of  $0 \sim 1.2 \text{ N/mm}^2$  with a sensitivity of  $11 \text{ cm}^{-1}/\text{MPa}$ . The sensitivity is about a hundred times larger than the coefficient reported under hydrostatic pressure. The barycenter energy of the emission band of a phosphor can be used to indicate stress and/or temperature change, producing a precision beyond the spectroscopic resolution limit. The sensing functions obtained from the barycenter shift are superior to those from peak shifts. The stress/temperature dependent barycenter shift of the observed fluorescence bands of YAG:Ce were similar in magnitude. Mechanisms involving the nephelauxetic effect, crystal field effect and site symmetry of dopant ions are explored as explanations of the experimental phenomena.

**Keywords** Stress · Temperature · Sensing · Fluorescence

## Introduction

It is well known that certain micro-force sensors make use of changes in the fluorescence emitted from specific materials to measure both applied pressure [1, 2] and stress [3]. The changes in fluorescence are often attributed to a shortened bond length between atoms in a strengthened crystal field, resulting in a shift in the associated energy levels. For such sensing applications, a wavelength shift at the intensity maximum of the emission peak(s) of a phosphor is normally used as the sensing element. However, because the scale of the shift is always small, the fluorescence intensity needs to be strong to achieve an acceptable signal-to-noise ratio. Even so, considering that it is desirable that the peak shift should be significant upon applied pressure or stress, there are not many choices for the active material. Ruby ( $\text{Al}_2\text{O}_3:\text{Cr}^{3+}$ ) is a popular active material whose sharp emission peak(s) facilitate distinguishing a slight shift of the peak. The fluorescence of  $\text{Cr}^{3+}$  has found wide application in hydrostatic pressure detection in a diamond anvil cell [4] and in residual stress sensing [5]. However, when using the fluorescence method only high pressure or stress (over hundreds MPa) can be retrieved with relatively high precision.

Some rare-earth doped phosphors have very high quantum efficiency, e.g. the well-known yellow phosphor, YAG:Ce. For this phosphor the parity allowed 5d-4f inter-configurational transitions of  $\text{Ce}^{3+}$  are highly sensitive to the change of environmental factors because the d-electrons are in the outer shell. As a consequence the phosphor is known to exhibit pressure-dependent fluorescence which may be attributed to the change of the Ce-O bond length [6]. Therefore, the radiative transitions of  $\text{Ce}^{3+}$ -doped phosphors are expected to be sensitive to changes in stress and/or temperature which lead to similar Ce-O bond changes in the lattice of the matrix material. The emission spectrum of YAG:Ce reveals two highly

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overlapping bands. The application of high pressure drives the two bands to shift towards a lower energy with almost the same piezo-spectroscopic coefficient [6, 7]. Determining the peak position of the resultant broad band is imprecise, and as a consequence, it is difficult to accurately extract shifts in the peak position particularly at a low pressure or under small magnitudes of applied stress. This may explain why YAG:Ce has not been used previously as a probe for applied strain/stress of low magnitude.

The term “barycenter” is most often used in astronomy to describe the location of the center-of-mass of a many-body system. However, it has also come to be used to describe a weighted mean position of fluorescence peaks, particularly, as is the case here, where there is a broad spectral feature masking a number of underlying peaks [8–10]. In this article, it is shown that the barycenter of an emission band functions well as an alternative to the peak wavelength to indicate the stress change for low magnitudes of stress. An extra advantage is that such a sensing method does not require the measurement spectrometer to have a high resolution; an important consideration in keeping instrumental costs and dimensions to a minimum. In this work YAG:Ce has been used to illustrate the sensing technique. The technique described was also applied to temperature sensing, as the same physical effect is evident in the coordination change of  $\text{Ce}^{3+}$  caused by either stress or temperature.

## Experimental Arrangement

The sample precursor was a mixture of a YAG:Ce phosphor powder (Keheng™ Y4454) and a silicone resin with a mixing ratio of 1:5 by weight. Air bubbles were removed from the mixture under vacuum and then cured in air. The resulting compound was trimmed into a slice ( $6.0 \times 7.0 \times 1.5 \text{ mm}^3$ ). During the piezo-spectroscopic analysis, compression was applied along the thickness direction. The sample was sandwiched between a glass slide and a push force gauge, as shown schematically in Fig. 1. The resolution of the gauge was 0.25 N, and the measurement range was 0–50 N. For the temperature-dependent test, the phosphor powder was used unadulterated to avoid any possible influence of the resin material. In practice, no notable evidence of the resin's influence on the phosphor fluorescence was found in this work or has been reported elsewhere, while the silicone material is widely used in white LEDs as conformal coating on the wavelength-converting phosphor(s).

A diode laser with a peak wavelength of 405 nm was used as the excitation light source. All emission spectra were measured with an AvaSpec-2048TEC-USB2 fiber-optic spectrometer. The finest resolution of the spectrometer is 0.5 nm at the wavelength 500 nm ( $20 \text{ cm}^{-1}$  at the wavenumber  $20,000 \text{ cm}^{-1}$ ). A dye filter, located up beam of the

spectrometer, removed scattered excitation light from the recorded spectra. The phosphor's structure has been confirmed on a D8ADVANCE X-ray diffractometer, using a Cu  $K_\alpha$  X-ray source.

## Results and Discussions

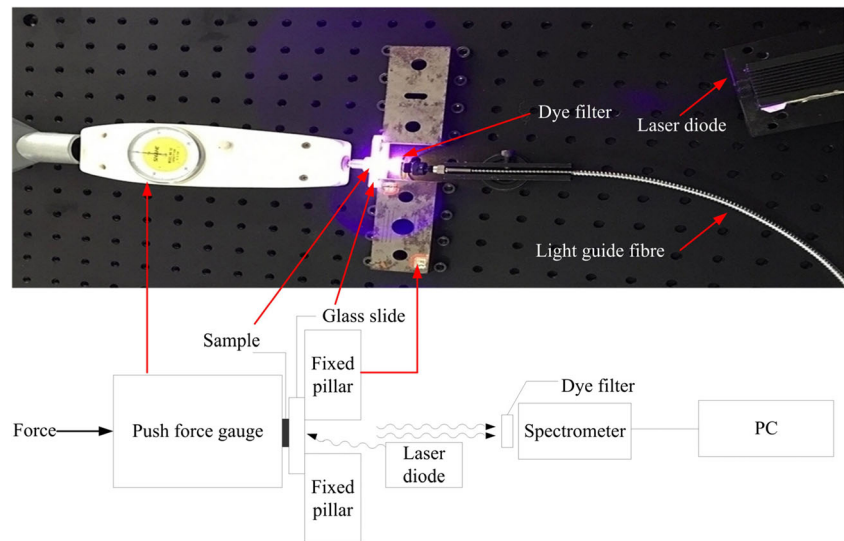
The X-ray diffraction (XRD) pattern of the phosphor powder sample is shown in Fig. 2 and accords with PDF (powder diffraction file) No. 88–2048, confirming that the phosphor has a garnet structure. The resulting emission spectra (Fig. 3) show fluorescence typical of  $\text{Ce}^{3+}$ -doped YAG. No obvious difference of the spectra features between the powder sample and the composite sample was found. At very low temperature, a spectrometer with fine resolution may directly distinguish two peaks at about 536 nm and 567 nm, respectively, in the emission spectrum of YAG:Ce [11]. Both emission bands are electron transitions from the lowest 5d level to 4f multiplets ( $^2F_{7/2}$  and  $^2F_{5/2}$ ) of  $\text{Ce}^{3+}$ .

The two wide fluorescence emission bands of YAG:Ce overlap to such an extent that stress-dependent changes are almost indistinguishable, as is evident in Fig. 3, leading to difficulties in locating precisely any changes to the peak position of the amalgamated band. For example, to observe a pressure-dependent peak shift in the fluorescence emitted from the phosphor, a pressure as great as tens of kilobar (several GPa) is required [7]. Accordingly, a high-resolution spectrometer would be obligatory for the measurement to be successful. When a low level stress is applied to the phosphor, it is not possible to observe any shift in the peak position for the emission spectra, as indicated by the left inset of Fig. 3. Likewise multi-peak fitting cannot be successfully used to reveal a trend in the relationship between the peak wavelength and the applied stress.

However, there is evidence in the spectra (Fig. 3) corresponding to changes in the applied compressive force. On the high energy side of the merged emission bands of  $\text{Ce}^{3+}$ , the band edge is observed to be shifting as shown in the right inset of Fig. 3 while on the other side of the band, the successive fluorescence curves essentially overlap each other. This observed shift is normally regarded as being in accordance with the experimental features of the nephelauxetic effect and the change of the crystal field strength [10, 12]. The expected manifestation of the nephelauxetic effect is for the barycenter energy of a rare-earth ion's energy levels to shift, characterizing a change in the coordination bond length of the ion. In this paper further analysis of the emission spectra is based on the observation of the barycenter shift of the emission band.

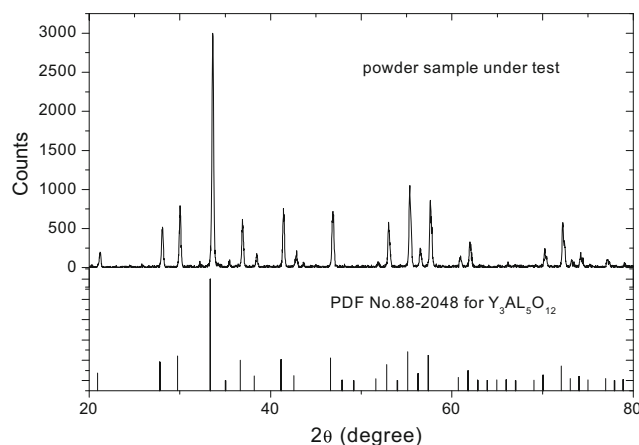
The nephelauxetic effect seeks to describe the size of the electron cloud around the central ion which increases due to the transfer of electron density to a bonding molecular orbital.

**Fig. 1** Schematic of the experimental setup for measuring stress-dependent fluorescence



Experimentally the effect can be seen as a shift of the absorption or emission bands caused by an environmental change resulting in changes to the fluorescence spectrum [12]. Spectral changes can therefore be linked to the environmental change and consequently the potential application as a sensing element is realized. For example, for the  $4f^n-4f^n$  transitions of  $\text{Eu}^{3+}$ , a linear relationship between the barycenter energy and temperature has been reported [9]. As for YAG:Ce, compression onto the phosphor composite makes the average Ce-O distance shorter and consequently lower the barycenter energy of the 5d levels. Taking into account that the emission relates to the transition from the lowest 5d level of  $\text{Ce}^{3+}$ , a strengthened crystal field will also contribute to a red shift of the emission band barycenter via increasing the splitting of the 5d levels.

Considering that the 4f electrons are shielded by the 5d electron shell, the ground 4f level(s) of  $\text{Ce}^{3+}$  is not expected to be greatly affected by environmental changes. Evidence for this lies in the fact that the two emission bands stay merged as

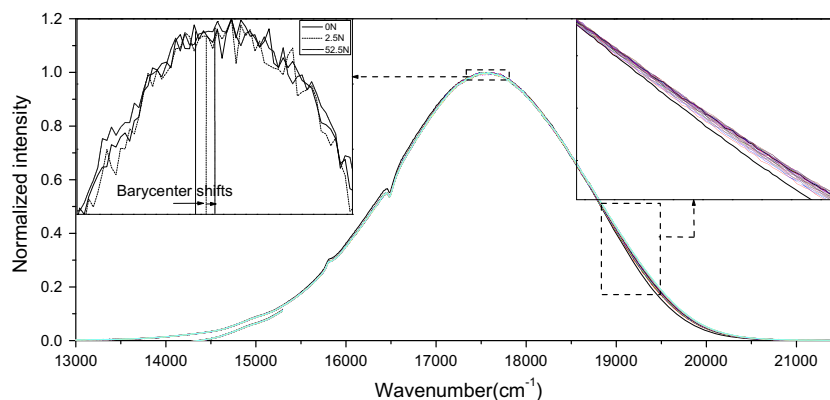


**Fig. 2** X-ray diffraction pattern of the YAG:Ce phosphor powder

one observable band, not separating under normal pressure [6, 7] or stress (Fig. 3). In this circumstance the separation width of the two sub-bands may be equated to the splitting width of the 4f ground level, which is determined by the crystal field strength. As mentioned above, multi-peak fitting cannot produce a self-consistent monotonic change of the separation width as the applied compressive force is increased, and such an analysis was not performed in this work. The compression-dependent shift of the  $\text{Ce}^{3+}$  emission band barycenter is shown in Fig. 4 and its magnitude is observed to be nearly equal to the shift of the 5d lowest level of  $\text{Ce}^{3+}$ . Hence by determining the shift in the barycenter of the emission bands, at various compression levels, the effect of compression becomes clearly observable in contrast to the very small changes to the peak positions of the two merged bands of  $\text{Ce}^{3+}$  emission. In fact, as shown in Fig. 4 and also in the left inset of Fig. 3, the fluctuations in intensity made any shift in the peak position almost indistinguishable, even though the signal-to-noise ratio of the spectra is relatively high at about 700:1 (calculated with the peak-to-peak baseline noise and the fluorescence peak intensity from the real spectra in this study).

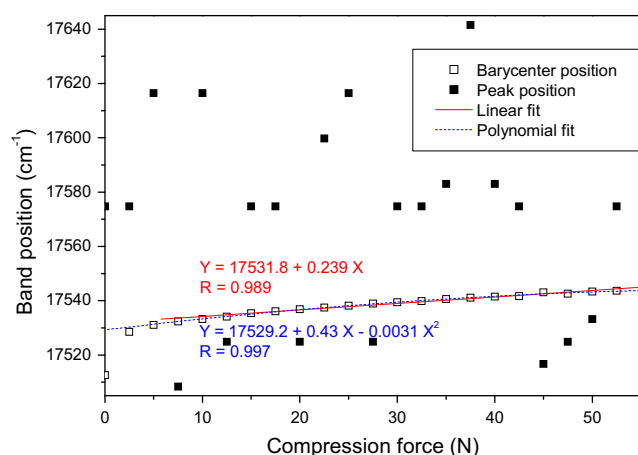
Figure 4 suggests a near linear sensing region for part of the range of applied compressive force. The line gives a piezospectroscopic coefficient of about  $11 \text{ cm}^{-1}/\text{MPa}$  (compression over the sample area  $\sim 42 \text{ mm}^2$ ). This shift coefficient is of the order of one hundred times larger than the reported hydrostatic pressure coefficient of the YAG:Ce emission peaks [7]. In addition the direction of the shift is the reverse of what is expected. As for the first datum point which is free of external stress loading, neither the linear nor the polynomial fitting curve are able to accommodate it, most probably implying the existence of residual stress in the composite sample. The measured resolution is less than  $0.06 \text{ N/mm}^2$  ( $0.06 \text{ MPa}$ ,  $2.5 \text{ N}$  over  $42 \text{ mm}^2$  surface area), with a barycenter energy shift of  $0.6 \text{ cm}^{-1}$ . This is better than the spectrometer resolution

**Fig. 3** Emission spectra of the composite sample under varying magnitude of compressive force. The spectra have been normalized to the peak intensity. Selected portions are magnified in the insets



( $20 \text{ cm}^{-1}$  at  $500 \text{ nm}$ ). That is to say, the sensing function performs well for applications of stress measurement at a very low magnitude of stress. Further, the sensing phenomenon is contrary to the prediction of the nephelauxetic effect and crystal field theory which would expect to present a red shift.

For practical applications, it is not essential that the measurement be related to the nephelauxetic effect when retrieving and/or applying the barycenter of fluorescence peaks/bands. Notwithstanding the physical meaning or any ambiguity of the stress sensing mechanism, the above experimental analysis of stress-dependent spectra has shown the advantages of using the barycenter technique. While the method is compatible with traditional peak-locating operations (as both characterize the band position) the resolution of the barycenter technique was shown to be superior to the resolution limit of the spectrometer used in the measurement. Therefore a very small stress magnitude is able to be distinguished by using the barycenter technique. To our best knowledge, it is the first time that a low stress (several KPa) has been precisely measured via a fluorescence method. Particularly there have not been any similar reports on the stress-dependent properties of  $\text{Ce}^{3+}$ -doped garnet family of materials.



**Fig. 4** Barycenter energy of the emission band of the composite sample under varying compressive force

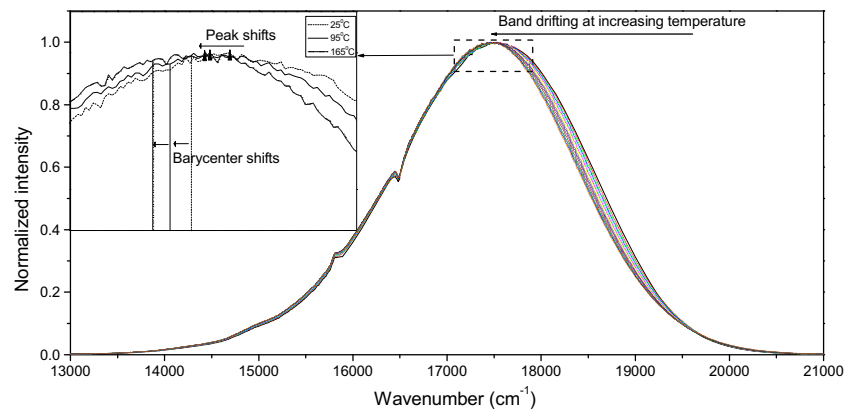
The unpredicted shift direction remains unexplained. Considering that the lanthanide-ligand distance is both temperature- and stress-dependent, an investigation of the temperature-dependent fluorescence (Fig. 5) was undertaken in an attempt to clarify the piezo-spectroscopic ambiguity. Any change in the barycenter energy caused by different environmental parameters can be considered as having the same coordination circumstance dependence [9].

Consistent with the unpredicted result with applied stress reported above, Fig. 5 shows temperature related shifts in the peak position opposite in direction to that expected by recourse to the nephelauxetic effect and crystal field theory. A thermally expanded lattice constant means weaker electron cloud repulsion and also a weakened crystal field. These effects should raise the lowest 5d level of  $\text{Ce}^{3+}$ , showing a blue-shifted emission band in direction contrast to the experimental result.

By way of exploration, there are other operations on the material that can vary the lattice constant and consequently influence the resultant fluorescence phenomena potentially in a way that is comparable with our observations. Such lattice expansion or shrinkage could be a result of the heat treatment temperature on sample phosphors [13, 14] or an effect of the nanoscale sample particle size [15, 16]. Alternatively there may have been  $\text{Y}^{3+}$  substitution by  $\text{Gd}^{3+}$  [17–23] or  $\text{Tb}^{3+}$  [24] or again  $\text{Lu}^{3+}$  [25, 26]; perhaps there was a different doping concentration of  $\text{Ce}^{3+}$  [27–29]. Even though these articles reported very similar wavelength shifts of the emission band maximum, the mechanism of such a lattice effect on  $\text{Ce}^{3+}$  emission were ambiguously allocated to strengthened or weakened crystal field in most of the articles. Tanner et al. have pointed out similar conflicts in various publications when they reassigned energy levels of  $\text{YAG:Ce}$  [30].

There are more conjectures that may be related to the shift of the emission of  $\text{YAG:Ce}$ . The garnet structure possesses several types of positions for the accommodation of dopant ions [31, 32]. Dopant pairs/clusters can possibly produce lower energy sites in the  $\text{Lu}_3\text{Al}_5\text{O}_{12}$  matrix and then the emission spectra show a strong red shift [33]. However, it is common

**Fig. 5** Temperature-dependent emission spectra of the phosphor powder sample, normalized to the peak intensity. A selected portion is magnified in the inset

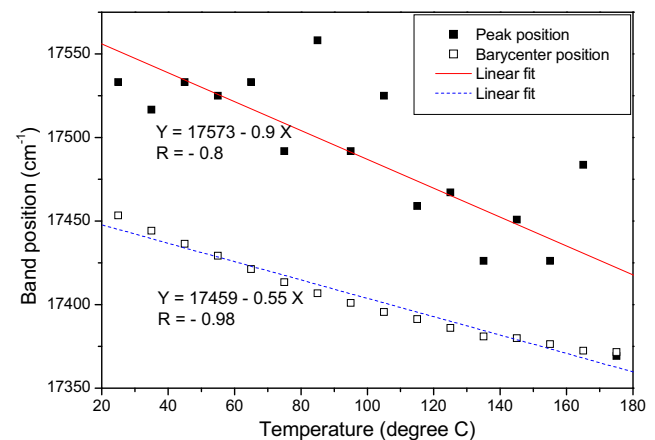


that  $\text{Ce}^{3+}$  replaces  $\text{Y}^{3+}$  in the YAG material but not in other positions if the synthesizing temperature is not too high [30, 34]. Moreover, in a certain material with fixed doping concentration, the dopant ions are not able to re-distribute reversibly under the influence of temperature or stress. Then the above findings do not reveal the major mechanism of the observations reported in this work. Neither do other speculations, such as re-absorption processes [35] or a modified crystal field theory [36]. Considering that the major mechanism of the observed shifts should be of the coordination circumstance of the central  $\text{Ce}^{3+}$  ion and nothing else, it was determined that an altered site symmetry of the central ion should be taken into account.

Andriessen and co-workers have shown that lattice deformation leads to a significant shift in  $\text{Ce}^{3+}$  ion fluorescence wavelength [37]. More reports [38–41] suggest that deviation from cubic symmetry around the  $\text{Ce}^{3+}$  ion results in an increase in the maximum emission wavelength, while yttrium aluminum garnet provides a distorted cubic local circumstance to the dopant [34]. Besides the radius of the dopant ion, environmental parameters such as temperature and stress can produce a similar distortion. Anisotropic thermal expansion of the matrix YAG material drives the Ce-O cell microstructure distortion. On the other hand, stress can shape the microstructure to achieve a higher or lower symmetry, depending on the stress direction and scale. On average, for a phosphor powder-resin composite, compression should slightly diminish the difference of Ce-O distances, meaning an increasing symmetry. Lower point symmetry of  $\text{Ce}^{3+}$  allows a significant modification of the separation gap between the lowest two 5d levels, namely the two  $^2\text{E}$  levels of  $\text{Ce}^{3+}$  in a garnet structure [38–41]. Both the temperature influence and the compression effect of YAG:Ce fluorescence can imply different distortions of the solid lattice. The distortion influence can be so important that the crystal field effect is more than being compensated. Only when the change of the coordination circumstance around  $\text{Ce}^{3+}$  is isotropic, e.g. under a hydrostatic pressure, the coordination symmetry issue is no longer effective. This consideration can explain why the

observed stress and temperature effects in the present experiment are different to those reported in previous publications [6, 7], whilst the phenomena is more complicated than the prediction of the nephelauxetic effect, crystal field theory, and the Jahn-Teller effect, e.g. the remaining problem in multi-peak fitting.

As well as the usage of the phosphor-resin composite as a stress gauge, the temperature-dependent monotonic shift of emission band (Fig. 5) can be used for temperature sensing. Influences of temperature on the position of both the band peak and barycenter energy are shown in Fig. 6. The trends in the data for each method of analysis are similar; however the barycenter shifts indicate a much smaller uncertainty in the determination of the sensitivity ( $(-0.55 \pm 0.03) \text{ cm}^{-1}/^\circ\text{C}$  compared with that for the position of the band peak ( $-0.86 \pm 0.17) \text{ cm}^{-1}/^\circ\text{C}$ ). The form of barycenter sensing line is well defined but not yet understood. To enable a comparison with the peak center technique, the barycenter sensing line has been fitted with a linear function. The correlation coefficient for the linear fitting of the barycenter sensing is  $-0.98$ , suggesting that the linear fit is acceptable and applicable. This improvement in measurement precision is a consequence of the



**Fig. 6** The temperature-dependent position of peak energy (solid line) and barycenter energy (dashed line) of the powder phosphor's emission band



analyzing methodology itself as when determining the barycenter energy, the total area of the emission band is integrated first and then the barycenter position divides the area equally. In this way the operation of integration eliminates the random noise of the measurement.

In this article, a shift in the emission band barycenter of  $\text{Ce}^{3+}$  indicates more than the change of Ce-O distance, either in the stress effect or temperature-dependent spectra. Reasonably, we may look forward to more sensing applications of the barycenter method in other situations with or without change of point symmetry at the active ion position.

## Conclusions

In conclusion, this article has demonstrated that fluorescent materials, in particular YAG:Ce, are available for stress and temperature sensing by carefully monitoring the position of the emission band(s). Sensing functions have been retrieved successfully and beyond the expectation of normal peak analysis when the barycenter energy was employed to characterize the band position. The barycenter energy of emission band(s) can be used as a sensing signal, while the actual operation of a practical device does not require consideration of the underlying physical effect. The barycenter energy provides a relatively fine resolution and high precision to the sensing method. This result is important for sensing applications at low pressure/stress levels in contrast to the “classic” peak shift method that has already proven to be successful for relatively higher stress amplitudes.

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